

THE TRIO-01 EXPERIMENT: IN-SITU TRITIUM-RECOVERY RESULTS*

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THE TRIO-01 EXPERIMENT: IN-SITU TRITIUM RECOVERY RESULTS*

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ABSTRACT

The TRIO-01 experiment is a test of in-situ tritium recovery from γ -LiAlO₂ with test conditions chosen to simulate those anticipated in fusion power reactors. A status report is presented which describes qualitatively the results observed during the irradiation phase of the experiment. Both the rate of tritium release and the chemical forms of tritium were measured using a helium sweep gas which flowed past the breeder material to a gas analysis system.

1. INTRODUCTION

The TRIO-01 experiment is a comprehensive test of in-situ tritium recovery from a miniaturized solid breeder blanket assembly. The design and objectives of the experiment are described in a previous report.¹ Presented herein are key results and observations during the irradiation as well as a status report of work in progress.

2. FABRICATION AND CHARACTERIZATION OF γ -LiAlO₂ BREEDER MATERIAL SPECIMENS

LiAlO₂ powder was prepared by mixing stoichiometric quantities of Al₂O₃ and isotopically depleted ($\sim 0.5\%$ ⁶Li) Li₂CO₃ in a water slurry. The slurry was spray dried, and then calcined in air at 800°C for eight hours. The microstructure of the pellets is shown in Reference 1. Small grains 0.1 μ m, were combined into

roughly spherical agglomerates ~ 50 μ m in diameter. The agglomerates were sintered together, providing a matrix with large pores to facilitate gas phase transport of the tritium released. This type of microstructure with a bimodal pore distribution is considered advantageous for tritium release as shown in STARFIRE.²

The characteristics of the fabricated pellets are summarized in Table 1. X-ray diffraction showed that the material was phase pure γ -LiAlO₂. The ⁶Li isotopic content was determined to be $0.55 \pm .01\%$. Chemical analyses using emission spectroscopy and neutron activation showed that the LiAlO₂ had cation impurity levels <100 ppm. The chloride level initially was high, about 2000 ppm. Heat treatment for 20 hours at 830°C reduced the chloride

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TABLE 1
 γ -LiAlO₂ breeder pellets - characteristics

Microstructure:	Bimodal pore distribution having grains 0.1 μ m diameter and agglomerates ~50 μ m diameter. Smear density ~65% of theoretical.
Material:	Phase-pure γ -LiAlO ₂ .
Contaminants:	Cations <100 wppm Halides < 50 wppm H ₂ O \leq 0.4 wt % CO ₂ \leq 0.4 wt %
Pellet Size:	Hollow cylinders, 25.4 mm O.D. x 15.9 mm I.D. x ~12.7 mm high. Seven pellets - total weight 12.9 g.
Isotopic Content:	Depleted, 0.55 \pm .01% ⁶ Li.

level to 500 ppm. A final baking of the pellet for 17 hours at 1050°C reduced the chloride content to 15 \pm 5 wppm.

3. EXPERIMENTAL CONDITIONS

The irradiation phase of the experiment was conducted in the A2 core position of the Oak Ridge Research Reactor (ORR). The assembly, or capsule, contained pellets of γ -LiAlO₂ in the form of hollow cylinders. The γ -LiAlO₂ pellets were so arranged¹ that the sweep gas (nominally He/0.1% H₂) flowed past the inside surface of the pellets and then out to the gas analysis system³ where a number of analytical measurements were performed. The temperature was controlled by means of varying the He/Ar ratio of the flowing gap gas in the inner and outer capsules. Approximately 50 parameters (Table 2) were continuously monitored and recorded during irradiation. This information is presently being analyzed.

The test matrix (Table 3) consisted of 33 runs at different temperatures and sweep gas flow conditions (composition, flow rate). The nominal gas composition was He/0.1% H₂ flowing at 100 cc/min. Other compositions were 100% helium and He/0.2% O₂. The effect of sweep gas flow rates was examined at 300 cc/min and 30 cc/min. The total irradiation fluence was approximately 3 x 10²¹ nvt and the lithium burnup was ~0.2%.

4. EXPERIMENTAL RESULTS

Significant results and observations to date are reported. These results will be completed after post-irradiation examination is done and a final analysis is performed.

4.1 Tritium release

Preliminary experimental results from the first nine runs (Table 3) are shown in Figure 1. The dark line represents the response of the tritium monitor which

TRITIUM LEVELS IN SWEEP GAS, Ci/m³

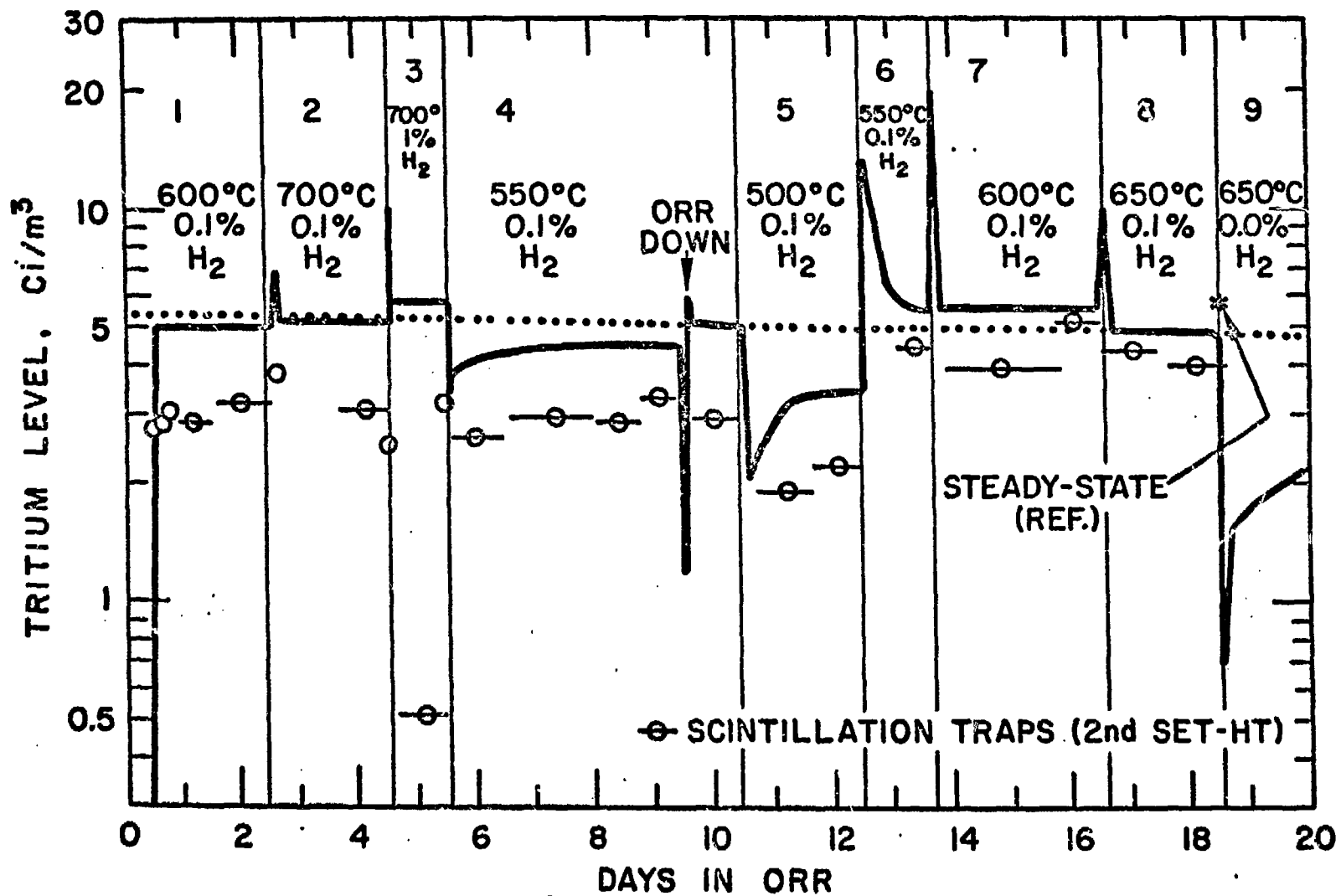


FIGURE 1

Preliminary tritium release results from the irradiation of γ -LiAlO₂ in the TRIO-01 experiment.

TABLE 2
Parameters monitored in the TRIO-01 experiment

Temperature	<ul style="list-style-type: none"> • The outer and inner diameter of the breeder pellets in two orientations designated east and west - ten thermocouples. • Seven reaction beds (oxidation, reduction, and getter in the gas analysis system) - seven thermocouples.
Neutron Flux	<ul style="list-style-type: none"> • Self-powered neutron detectors on the outside of the assembly for a continuous measure of thermal flux incident on the capsule. • Dosimeters located inside and outside the capsule to provide fluence data on the γ-LiAlO₂ pellets. • Reactor power - continuous monitor.
Tritium	<ul style="list-style-type: none"> • On-line monitors to record tritium in HT form. • Traps to collect HTO and HT forms. • Gas samples for mass spectroscopy. • Collection of tritium permeating into gap gas. • Tritium levels in secondary containment - monitored continuously. • Tritium level in laboratory air - monitored continuously.
Other Nuclide	<ul style="list-style-type: none"> • Other radionuclides, measured by Ge(Li) detector.
Gas Flow	<ul style="list-style-type: none"> • Pressure and flow rate of sweep gas and gap gas monitored.

senses the molecular (HT+T₂) tritium level. Corrections for meter calibration, pressure, and flow are in progress to provide quantitative values. The dotted line represents an estimate of the tritium generation rate. Final values will be those obtained from dosimetry. The small barred circles represent integral tritium which was measured in scintillation traps. The results in Figure 1 are representative of those obtained in all 33 runs. Individual results are discussed below.

• Recoil effects: When the breeder assembly was exposed to neutron at low power (3-30 kW and up to 12 MW), a measurable response was noted in the tritium monitors. Breeder temperatures were 30-200°C respectively. It was estimated, therefore, that ~5% of the tritium generated during these low temperature tests was released by recoil.

• Temperature effects: The tritium release rate at a nominal 600°C with a sweep gas composition of He/0.1% H₂ appeared to reach steady state within a few hours. An increase in the nominal breeder temperature resulted in a small positive spike which represented the difference between the tritium retained at a nominal 600°C versus that retained at a nominal 700°C. A decrease in temperature to 550°C resulted first in a negative spike which represented tritium accumulation at the lower temperature. Equilibration was slow, requiring more than three days to build a steady state tritium profile. A further decrease in temperature to 500°C resulted again in a negative spike. Temperature cycling resulted in reproducible behavior.

• Sweep gas composition: Removal of the hydrogen additive from the sweep gas has a

TABLE 3
Test matrix for TRIO-01

Run No.	Time (Days)	Nominal ^a Temperature (°C)	Changes in Nominal Sweep Gas Composition
1	2	600°	-
2	2	700°	-
3	1	700°	He/1.0% H ₂
4	5	550°	-
5	2	500°	-
6	1	550°	-
7	3	600°	-
8	2	650°	-
9	6	650°	100% He
10	1	400°	100% He
11	1	500°	100% He
12	4	550°	100% He
13	5	600°	100% He
14	2	700°	100% He
15	1	700°	-
16	1	700°	300 cc/min
17	3	700°	30 cc/min
18	1	700°	-
19	1	650°	-
20	4	650°	He/0.2% O ₂
21	2	650°	-
22	1	600°	-
23	3	550°	-
24	1	600°	-
25	2	500°	-
26	3	550°	-
27	7	525°	-
28	7	500°	-
29	3	480°	300 cc/min
30	4	480°	-
31	2	500°	-
32	2	550°	-
33	3	650°	-

^aAverage temperature on west side. Coldest temperature is ~100° lower, hottest temperature is approximately 50° higher.

negative effect upon tritium release (Run 9 vs. Run 8). In a later test (Run 20), addition of oxygen to pure helium did not appear to enhance release rates.

• Fluence effects: Tritium release rates did not appear to have significantly changed from the beginning to the end of irradiation.

4.2 Amount and form of tritium collected

The total amount of tritium collected was ~35 Ci. Of this total, ~5% represented tritium permeation into the gas annular gap. The majority of the tritium collected was present as HT and T₂ (99%).

4.3 Temperature control

The temperature of the breeder was monitored by thermocouples at ten separate locations as a function of radius (inside vs. outside); axial position (along the length); and geographic direction (east vs. west). The radial temperature gradients were an average of 100°, and longitudinal gradients were <10°. There was a side-to-side temperature difference, the east side being 50° colder than the west side. Thus, for a given nominal temperature, some breeder material was ~50°C hotter and some was ~100°C cooler.

The east side was postulated to be colder owing to asymmetry in gap dimensions. Recent PIE results showed that the inner capsule was leaning directly to the east side, thereby supporting the postulate.

4.4 Thermal conductivity effects

During irradiation, about 0.2% of the lithium in the sample was consumed. This lowered the heating in the capsule from about 560 W to 510 W, or about 10%. The radial temperature gradient systematically decreased during irradiation, being 10% lower at the end of irradiation. Since the thermal conductivity is proportional to the ratio of the heat

transport rate divided by the radial temperature gradient, it appears that the thermal conductivity did not change significantly during the experiment.

4.5 Radionuclides detected

The trace levels of contaminants such as Na, K, and U led to production of volatile radionuclides which were detected by γ -ray spectroscopy during the experiment. The primary species detected were F, Ne, Cl, Ar, Kr, Rb, Xe, and Cs. It was noted that the activities were very strongly dependent upon the breeder material temperature, release rates were faster at higher temperatures. This topic will be addressed later.

4.6 Design of gas analysis safety system

The gas analysis system, with two levels of containment to protect workers, worked very well. The tritium levels in the sweep gas were of the order of 5 Ci/m^3 (six orders of magnitude above maximum levels of HTO in air). The levels in the glove box were $\sim 100\text{--}1000 \text{ }\mu\text{Ci/m}^3$. Levels in the room were $< 1 \text{ }\mu\text{Ci/m}^3$ during the course of the experiment.

5. WORK IN PROGRESS

Quantitative analysis of the data requires the results from post-irradiation examination (PIE) as well as normalization of all data. PIE will consist of 1) residual tritium content of the breeder material, 2) ^6Li burnup, 3) dosimetry, and 4) characterization of the irradiated breeder specimens.

6. CONCLUSIONS

Although analysis of the data has not been completed, a number of conclusions can be made. 1) Tritium release and tritium recovery has been demonstrated for a miniaturized solid breeder blanket

assembly. 2) Thermal-hydraulic and heat transfer performance by controlled variation of temperature using a gas gap has been demonstrated. 3) The recovery of tritium from the helium sweep gas stream has been demonstrated. 4) Radioactive species are present in the sweep gas. 5) For LiAlO_2 , 5% of the tritium recovered had permeated through the primary cladding to the gap gas. 6) Addition of hydrogen to the purge gas appears to enhance tritium release. 7) Addition of oxygen to the purge stream does not enhance tritium release. 8) The containment systems performed well in protecting workers from high levels of tritium.

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